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Project Summary

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Metal Particulate Emissions from Stationary Sources

Volume 1. Standard Sampling and Analysis Method

A program was undertaken to develop reliable methods for measuring trace elements in emission streams. This program concerns a sampling and analysis method for arsenic (As), cadmium (Cd), chromium (Cr), cobalt (Co), manganese (Mn), nickel (Ni), lead (Pb), selenium (Se), and vanadium (V). Based upon a review of the literature, atomic absorption spectrophotometry was selected as the analysis method for all metals. To approximate a stationary source, a simulation system consisting of a fuel oil combustion chamber and stack with sampling ports was constructed. Known amounts of metals were introduced to the system as organometallic additives to the fuel. Flue gas sampling was carried out simultaneously with two trains permitting a direct comparison of changes in configuration, impinger solutions and sample recovery procedures. Based upon replicate experiments, precisions of 7 to 11 percent were obtained for all elements except As (15 percent) and Se (21 percent). Accuracies of Co, Ni, Mn, Cd, Cr and V were within 15 percent at the 100 µg level, with Pb being 31 percent high and As and Se being low by 32 and 48 percent, respectively.

This Project Summary was developed by EPA's Environmental Sciences Research Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

The objective of this work under EPA Contract No. 68-02-1219, is to develop reliable methods for sampling and analysis of potentially hazardous metal species present at trace levels in emission streams.

Because of their submicron particle size, typically reported to be in the range of 0.1 - 1 µm, collection by present control systems is not very efficient.

This report describes the development of standard sample collection and analysis methods for As, Cd, Cr, Co, Mn, Ni, Pb, Se and V, and an evaluation of the promulgated test methods 101 and 104 for mercury (Hg) and beryllium (Be), respectively.

Conclusions

An evaluation of the collection and analysis of known levels of As, Cd, Co, Cr, Mn, Ni, Pb, Se and V (and, in some cases, Be) from stack emission samples leads to the following conclusions:

- A single stack emission sample can be analyzed for all metals of interest. The procedure uses a Method 5-type sampling train with a quartz filter and 0.1 M nitric acid wet impingers. Special glassware precleaning and train cleanup procedures are

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required. After a nitric acid digestion of the combined samples from the collection train, analysis for each element is carried out by atomic absorption spectrophotometry.

- Based on nine replicate experiments at sample levels of about 100 μg for each metal, sampling and analysis yields a precision of 7 to 11 percent for all elements except As (15 percent) and Se (21 percent). The following accuracies (expressed as percentages) were obtained:

Pb	+31
Cd, Cr, V	+13 to 16
Mn	+6
Co, Ni	-2 to -4
As	-32
Se	-48

- The poor recoveries for As and Se may be due to incomplete combustion (oxidation) of the organometallic additives for these compounds, as the hydride evolution method is only suitable for the inorganic species of these elements.

- Analysis of Be at levels of 1 and 10 μg yielded good recoveries by Method 104 and by the sampling and analysis method described here for other metals.

- Evaluation of Method 101 for Hg yielded good recoveries at 1 μg level. For best accuracy, the average of at least 10 measurements of peak height should be used for analysis.

Recommendations

Further work is recommended with respect to simplifying the sampling train precleaning and sample recovery procedures and to improving the precision and accuracy for the analysis of As, Se and Pb.

Presently, the glassware is rinsed with concentrated HNO_3 during precleaning and sample recovery. It is recommended that the effectiveness of a 0.1 N HNO_3 rinse be evaluated. Besides reducing the risk of injury to operating personnel, this would reduce the number of samples to be collected. Furthermore, if shown to be suitable for sample recovery, the influence of 0.1 N HNO_3 on the stainless steel nozzle should be evaluated with a view to obviating the need to remove the probe liner for cleaning.

The low recoveries obtained for As and Se and the high recovery for Pb indicates problems with the sampling and analysis methods for these elements. Further studies should be conducted to identify the source of loss (or gain) for these elements. Specific areas to be considered include trace metal introduction into the combustion gas stream, losses within the combustion system, adequacy of the sampling train to collect these elements, recovery from the sampling train, sample workup procedure and analysis method.

This Project Summary was authored by Staff of the Center for Environmental Research Information, USEPA, Cincinnati, OH 45268.
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The complete report, entitled "Metal Particulate Emissions from Stationary Sources: Volume 1. Standard Sampling and Analysis Method," (Order No. PB 81-120 024; Cost: $11.00, subject to change) will be available only from:
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